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Extreme-Temperature Carbon- and Ceramic-Matrix Composite Nozzle Extensions for Liquid Rocket Engines

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Abstract

The United States (US) National Aeronautics and Space Administration (NASA) and its US industry partners are developing extreme-temperature composite nozzle extensions for a variety of cryogenic liquid rocket engine propulsion systems. Applications under consideration at the NASA Marshall Space Flight Center (MSFC) include launch vehicle (upper stage), in-space, and lunar lander descent/ascent propulsion systems. Composite material systems of interest include carbon-carbon (C-C) and carbon/silicon-carbide (C-SiC) composites, as well as modified versions of such composites (coatings, mixed matrices, oxidation inhibitors, etc.). Missions addressed include access to low Earth orbit, the lunar surface, and more distant destinations.

Cryogenic upper-stage and in-space liquid rocket engines are optimized for performance through the use of high area ratio nozzles to fully expand combustion gases to low exit pressures, increasing exhaust velocities. As a result of the large size of such nozzles, and the related engine performance requirements, composite nozzle extensions are being considered to reduce mass impacts. Currently, metallic and foreign composite nozzle extensions used on the Atlas V, Delta IV, Falcon 9, and Ariane 5 launch vehicles represent the state-of-the-art. Such extensions are limited to use at or slightly above 2000°F (1093°C). Materials under development by MSFC and its industry partners have the potential to operate at temperatures up to (or above) 4250°F (2343°C).

Marshall Space Flight Center efforts are aimed at (a) further developing the technology and databases needed to enable the use of composite nozzle extensions on cryogenic liquid rocket engines, (b) developing and demonstrating low-cost capabilities for testing and qualifying such nozzle extensions, and (c) advancing the US domestic supply chain for nozzle extensions. Through the Small Business Innovation Research (SBIR) and Small Business Technology Transfer (STTR) Programs, low-level internal MSFC research projects, and teaming arrangements with domestic industry partners, composite material design, development, hot-fire test, and evaluation efforts are progressing. Hot-fire engine tests of subscale hardware have been conducted using oxygen/hydrogen (LOX/H_2), oxygen/methane (LOX/CH_4), and oxygen/kerosene (LOX/RP-1) propellants. These engine tests at MSFC have enabled the evaluation of both heritage and state-of-the-art material systems, demonstrating the initial capabilities of the extreme temperature materials and their fabrication methods.

Recent, ongoing, and potential future work supporting cryogenic propulsion systems development will be presented. The composite nozzle extension technology and test capabilities being developed are intended to support both NASA and US Department of Defense requirements, as well as those of the broader Commercial Space industry.

Keywords: composite, carbon-carbon, ceramic matrix, nozzle extension, cryogenic, liquid rocket engine

Acronyms/Abbreviations

2D = Two Directionally-Reinforced
2.5D = Two and One Half Directionally-Reinforced
3D = Three Directionally-Reinforced
AA-2 = Ascent Abort Flight Number Two
ACC = Advanced Carbon-Carbon
ACM = Attitude Control Motor
ACO = Announcement of Collaborative Opportunity
AIAA = American Institute of Aeronautics and Astronautics
AL = Alabama
AM = Additive Manufacturing
C-C = Carbon-Carbon
C-CAT = Carbon-Carbon Advanced Technologies, Inc.
C/C-SiC = Carbon-Carbon/Silicon-Carbide
CH_4 = Methane
CMC = Ceramic Matrix Composite
CNE = Composite Nozzle Extension
COIC = COI Ceramics
C-SiC = Carbon/Silicon-Carbide
CTC = Concurrent Technologies Corp.
CVI = Chemical Vapor Infiltration
CVR = Chemical Vapor Reaction

DoD = Department of Defense
EUS = Exploration Upper Stage
FMI = Fiber Materials, Inc.
GE = General Electric
H_2 = Hydrogen
ICBM = Intercontinental Ballistic Missile
ICPS = Interim Cryogenic Propulsion Stage
ILT = Interlaminar Tension
IRAD = Internal Research and Development
IUS = Inertial Upper Stage
LAS = Launch Abort System
LCUSP = Low Cost Upper Stage Propulsion
LESS = Leading Edge Structural Subsystem
LLC = Limited Liability Corporation
LOX = Liquid Oxygen
META4 = Methane Engine Thrust Assembly for 4K-lb$_f$ thrust
MI = Melt Infiltration
MR&D = Materials Research and Design, Inc.
MSFC = Marshall Space Flight Center
NASA = National Aeronautics and Space Administration
NDE = Nondestructive Evaluation
NGIS = Northrop Grumman Innovation Systems
NTP = Nuclear Thermal Propulsion
PAM = Payload Assist Module
PAN = Polyacrylonitrile
PIP = Polymer Infiltration and Pyrolysis
PSI = Physical Sciences Inc.
QM-1 = Qualification Motor Number One
RMI = Reactive Melt Infiltration
RP-1 = Kerosene or Rocket Propellant Number One
SBIR = Small Business Innovation Research
SLS = Space Launch System
STTR = Small Business Technology Transfer
TA&T = Technology Assessment and Transfer, Inc.
TCA = Thrust Chamber Assembly
TRL = Technology Readiness Level
TS = Test Stand
US = United States
USA = United States of American

1. Introduction, Applications, and Background

The United States (US) National Aeronautics and Space Administration (NASA) and its industry partners are developing extreme-temperature composite nozzle extensions (CNE's) for a variety of rocket propulsion systems. Applications of interest to NASA, and especially the Marshall Space Flight Center (MSFC), include (a) launch vehicle (upper stage) liquid engines, (b) in-space liquid and nuclear thermal propulsion (NTP) systems, and (c) lunar/Mars lander descent/ascent liquid engines. Missions addressed include access to low Earth orbit, the lunar surface, and more distant destinations, such as Mars. Propulsion systems for such missions typically make use of cryogenic propellants and CNE's, which typically make use of carbon fiber reinforcement architectures in carbon and/or ceramic matrices. The primary carbon and ceramic matrix composite (CMC) material systems under consideration include carbon-carbon (C-C) and carbon/silicon-carbide (C-SiC) composites, as well as modified versions of such composites (that make use of coatings, mixed matrices, oxidation inhibitors, etc.).

This paper provides an overview of some of the research and development work that NASA and the US aerospace and composites industry are conducting to advance the technology readiness level (TRL) of CNE components. The goal is to mature the technology such that large-scale domestically-manufactured CNE's can be considered as viable candidates for use on US cryogenic liquid propulsion rocket engines. This technology is intended to support the needs of both the Commercial Space transportation industry and the US government – NASA and the US Department of Defense (DoD).

For NASA, CNE technology development is aimed primarily at satisfying the requirements of the Artemis Program (Space Launch System / Orion) and the Human Landing System Program, as well as the requirements of its Commercial Space partners (see Figs. 1 and 2). The first two flights of the Artemis Program will make use of the Boeing Interim Cryogenic Propulsion Stage (ICPS) and its ArianeGroup (France) polyacrylonitrile- (PAN-) based C-C nozzle extension [1,2,3]. The ICPS uses a single RL10B-2 liquid-oxygen/liquid-hydrogen (LOX/LH$_2$) engine that is the same as was previously used on all Delta IV Centaur Upper Stages. The RL10B-2 (and follow-on RL10C-1) is the only US liquid cryogenic engine that has flown with a C-C composite nozzle extension. After the Artemis-2 mission, the SLS will make use of the upgraded Exploration Upper Stage which will make use of four RL10C-3 engines (see Fig. 3).

Fig. 1. Space Launch System (SLS) Exploration Upper Stage (EUS) with RL10C-3 engines.

Fig. 2. One concept for a 2024 human lunar lander. Both descent and ascent stages are shown.

Fig. 3. Four RL10C-3 engines in SLS EUS configuration. Each nozzle extensions consists of a fixed (non-translating) carbon-carbon “A” plus “B” cone set.

Upper-stage and in-space liquid rocket engines are optimized for performance using high area ratio nozzles in order to fully expand combustion gases to low exit pressures, thus increasing exhaust velocities. The large size of such nozzles, and their related engine performance requirements, makes composite nozzle extensions highly desirable for reducing the overall engine system mass. The metallic and foreign composite nozzle extensions used on the Atlas V, Delta IV, Falcon 9, and Ariane 5 launch vehicles represent the current state-of-the-art. These nozzle extensions are limited to use at or slightly above 2000°F (1093°C). Materials presently under development have the potential to enable operating temperatures of up to (or above) 4250°F (2343°C). Overall, the use of CNE’s is desired for a variety of reasons, including:

- The use of CNE’s enables approximately a 50% reduction in mass (weight) versus that of comparable metallic or ablative nozzle extensions.
- Using C-C CNE’s significantly improves thermal margins versus that of comparable metallic nozzle extensions. As uncooled metallic nozzle extensions are limited to temperatures of around 2000ºF (1093ºC) [4,5] using state of the art metals, CNE’s offer improved performance capabilities and efficiencies through greater thermal capabilities – increases of 500° to 1000ºF (278° to 556°C) are achievable, enabling upper use temperatures of 3000ºF (1649ºC). New and emerging composite materials may enable CNE designs that offer increases of up to 2000ºF (1111°C) , with upper use temperatures of 4250ºF (2343ºC) being possible.
- Substantial reductions in overall costs are possible with CNE’s when compared to metallic nozzle extensions and foreign composite nozzle extensions. Even greater cost and mass reductions may be possible if the regeneratively-cooled portion of the metallic nozzles can be shortened enabling the use of longer CNE’s.
- Finally, the potential use of state-of-the-art coatings and mixed and/or inhibited matrices may further increase the potential capabilities of advanced CNE’s and may lead to higher thermal performance.

Current efforts to advance composite nozzle extensions for liquid systems are based upon the technology developed under solid propulsion programs of the 1970’s and 1980’s, even though the requirements and operating conditions for cryogenic liquid engines are considerably different from those for solid rocket motors. Solid motor programs led to the development of uncoated C-C exit cones for (a) intercontinental ballistic missiles (ICBM’s), such as the Peacekeeper (see Fig. 4) and the Midgetman; and (b) solid motor upper stages, such as the Inertial Upper Stage (IUS) and the Star 48 Payload Assist Module (PAM). The only flight-proven coating for C-C components in the 1980’s was the silicon carbide coating system used on the Space Shuttle Orbiter’s wing leading edge structural subsystem (LESS) panels. During this time period, the ballistic missile and upper-stage flight programs experienced a number of problems with their solid motor exit cones, including processing variability and the in-flight loss of two Star 48 motors in 1984 [6]. This led to a reluctance to consider CNE’s for liquid engines. Additionally, the collapse of the Soviet Union in 1991 led to a greatly reduced US C-C industry, as the need for such materials dropped precipitously. In the 1990’s, the Delta III/IV Programs decided to use C-C composite nozzle extensions on the Centaur Upper Stage’s RL10B-2 engine. The selection was based upon consideration of a niobium-alloy (C-103), a HITCO Carbon Composites 2D (two directionally-reinforced) C-C, and an ArianeGroup 3D (three directionally-reinforced) C-C – the ArianeGroup material was chosen primarily due to weight considerations (versus the C-103 option) and, although solved, delamination concerns (versus the HITCO option). To date, the ArianeGroup

CNE’s for Delta IV Centaur Upper Stages have performed flawlessly [7].

Fig. 4. US Air Force Peacekeeper ICBM test flight. The Boeing led program included 50 test flights. Stage III of the missile had a C-C extendible exit cone.

With a lack of liquid engine application opportunities and the associated high costs of developing, qualifying, and certifying new nozzle extensions, there was insufficient impetus in the US to develop a CNE for an upper stage engine until the NASA Constellation Program (and subsequently the SLS Program) initially baselined a C-C nozzle extension for the J2-X engine. Ultimately, the Constellation/SLS J2-X engine development effort switched to a metallic approach because of a variety of cost, technical, and programmatic reasons [8]. However, the work performed for the J2-X engine program helped generate significant interest in CNE approaches. Additionally, more recently, the substantial growth in the number of launch vehicle, in-space, and lander applications has greatly increased opportunities for using CNE’s.

Acceptance of the potential significant advantages of CNE’s over metallic and ablative options has also improved significantly, with most systems under development now exploring CNE options. Since the J2-X CNE work ended, the range in types of propulsion system applications, composite component sizes, and composite materials available have all increased substantially. The range in high-temperature composite component sizes for rocket propulsion applications are represented by the Orion Launch Abort System (LAS) Attitude Control Motor’s (ACM’s) pintle valves and the Nuclear Thermal Propulsion (NTP) nozzle extensions (see Figs. 5 and 6, respectively). The Orion LAS ACM pintle valves have parts with dimensions in the 2-4 in. range, while a projected NTP CNE has an aft-end diameter of over 9 ft. (similar in size to that of the J2-X engine CNE). While not a liquid engine system, the Orion LAS ACM material is of a composite type similar to those being considered for liquid systems, specifically a carbon-carbon/silicon-carbide (C/C-SiC) material. The materials being explored for rocket propulsion applications are also similar to many of those that are under consideration for hypersonic vehicles (heat shields and hot structures for Earth, Mars, etc. entry, descent, and landing) and fusion energy reactors (tokamak first wall tiles) [9]. Thus synergies with non-propulsion uses offer additional means to advance the required technology.

Fig. 5. Orion LAS ACM Qualification Motor-1 (QM-1) test on 20 March 2019 at Northrop Grumman Innovation Systems (NGIS) facility in Elkton, Maryland. The system was also more recently successfully demonstrated with the Orion LAS Ascent Abort-2 (AA-2) flight test on 2 July 2019.

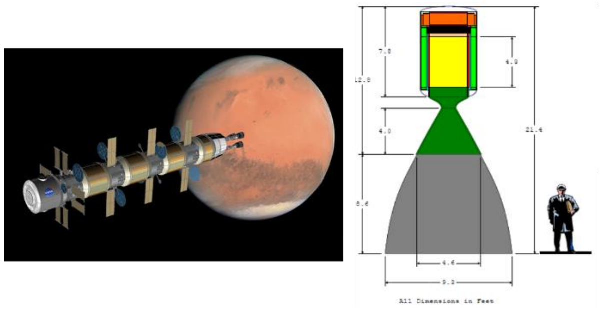

Fig. 6. (a) Conceptual design for a NASA NTP in-space system. (b) Pre-conceptual design schematic of a NTP system showing the size of a potential CNE – height = 8.6 ft., aft-end diameter = 9.3 ft. (this is very similar to that previously planned for the J2-X engine – height = 7.9 ft., aft-end diameter = 9.8 ft.).

To be fully flight qualified, more effort is required for composite nozzle extensions in the areas of material processing (including stable, reliable, sources for precursor materials), material characterization/databases, modeling capabilities, engine hot-fire testing, and viable paths to flight certification. Some of the recent and on-going work being pursued in these areas is presented below.

2. Summary of NASA-Funded CNE Materials Development Efforts

The recent surge in interest in using composite nozzle extensions, for a relatively wide variety of NASA, DOD, and Commercial Space propulsion systems, is due primarily because of the following:

- The mass (weight) reductions possible,
- The improved engine performance capabilities that are achievable,
- The potential for significantly reduced costs, as well as
- The rapidly expanding number and variety of potential commercial and exploration missions.

Under the Constellation Program's J-2X engine development effort, ArianeGroup was selected as the supplier of the carbon-carbon CNE in 2007 primarily because of the demonstrated success of the C-C nozzle extensions used on the Delta IV's RL10B-2 upper stage engines. Even though the J-2X Program eventually switched to a metallic nozzle extension, the composites development work accomplished stimulated significant interest in C-C nozzle extensions, including possible domestically sourced options. Progress since that time has largely continued under a variety of NASA SBIR/STTR (Small Business Innovation Research / Small Business Technology Transfer) projects, in-house NASA technology development tasks, and industry-led internal research and development (IRAD) efforts [10]. Thus, the state of CNE technology has continued to advance. With the recent acceleration of the NASA human exploration program under the Artemis Program, and the tremendously increased emphasis on both robotic and human lander systems (which includes NASA's Human Landing System Program), the Commercial Space industry is greatly facilitating the advancement of composite nozzle extension technology. As an example, Blue Origin, LLC (limited liability company) has just entered into an Announcement of Collaborative Opportunity (ACO) partnership with NASA's Marshall Space Flight Center and Langley Research Center to evaluate and mature high temperature liquid rocket engine nozzle material technologies for use on their commercial lunar transportation system, Blue Moon. There are several other non-public commercial space examples like this, further demonstrating the renewed interest by industry.

Companies involved in the development of domestic fabrication capabilities for high temperature composite nozzle extensions include those shown below. This is not all-inclusive list, but does include many of the companies that NASA has been working with to develop CNE technologies. Note: Those companies involved in the development of fiber, fabric, weaving technology, and other precursor materials are not shown in this list.

- Allcomp, Inc. – City of Industry, California
- Carbon-Carbon Advanced Technologies, Inc. (C-CAT) – Kennedale, Texas
- Exothermics, Incorporated – Amherst, New Hampshire
- Fiber Materials, Inc. (FMI) – Biddeford, Maine
- General Electric (GE) Aviation – Newark, Delaware
- HRL Laboratories, LLC – Malibu, California
- HITCO Carbon Composites, Inc. – Gardena, California
- Materials Research and Design, Incorporated (MR&D) – Wayne, Pennsylvania
- Northrop Grumman Innovation Systems (NGIS) – Magna and Promontory, Utah
- Northrop Grumman Innovation Systems COI Ceramics (COIC) – San Diego, California
- Physical Sciences Incorporated (PSI) – Andover, Massachusetts
- Plasma Processes, LLC – Huntsville, Alabama
- Technology Assessment and Transfer, Incorporated (TA&T) – Annapolis, Maryland
- Ultramet – Pacoima, California

A portion of the recent development work performed under NASA SBIR and STTR contracts, NASA MSFC in-house projects, and a NASA "Tipping Point" public-private partnership is presented below. P.R. Gradl and P.G. Valentine reported additional work in these areas previously in 2017 [10]. These development projects have concentrated primarily on two broad technology areas:

- Attachment technology concepts, and
- Materials fabrication and characterization methods.

2.1 Attachment technology concepts

Two projects are briefly discussed below:

2.1.1 "Regeneratively Cooled Ceramic Matrix Composite Nozzle Assembly for Reduced Weight"

Physical Sciences, Inc. (PSI) is leading this small business technology transfer (STTR) project with support from their research institution partner, Concurrent Technologies Corporation (CTC). The goal of the project is to develop actively-cooled, high-performance, lightweight nozzles and combustion chambers for upper stage and planetary descent/ascent oxygen-methane liquid rocket engines. The STTR Phase I contract, which ran from 27 July 2018 through 26 August 2019, made use of (a) PSI's expertise with carbon/silicon-carbide (C-SiC) ceramic matrix composite (CMC) materials, and (b) CTC's expertise with additively manufactured metallic structures. The

Phase I effort investigated and demonstrated potential techniques for fabricating C-SiC components with integrally-bonded metallic cooling passages. The C-SiC portion of the combined CMC/metallic components is intended to contain the hot combustion gases of a propulsion system and the metallic portion is designed to provide exterior cooling of the C-SiC to enable significant improvements in thermal capabilities and performance with substantial decreases in weight versus all-metal designs. Obtaining an intimate bond between the C-SiC composite and the metallic cooling structure is one of the key technical challenges being investigated.

2.1.2 "Domestically Available C/C for Hot Structure"

Materials Research and Design, Inc. (MR&D) is leading this small business innovation research (SBIR). The goal of the project is to demonstrate the manufacturability of domestically available, needled lyocell-based carbon-carbon materials for use in composite nozzle extension and exit cone applications. This Phase I project started on 19 August 2019 and runs through 18 February 2020. The MR&D led effort aims to demonstrate that combining the inherently desirable properties of the lyocell carbon fiber (i.e. "high" coefficient of thermal expansion, low modulus of elasticity) with a needling process results in a domestically available 2.5D (two and one half directionally-reinforced). CNE material (see Fig. 7) that has attractive properties for joining the C-C composite to metallic engine components. Lyocell-based C-C has properties very similar to those of the rayon-based C-C's used previously for solid motor rocket propulsion applications and the Space Shuttle orbiter wing leading-edge panels [11]. This six-month project will include a design and analysis trade study coupled with the fabrication and testing of nozzle extension subcomponents in order to demonstrate the viability of the CNE material for attachment concepts.

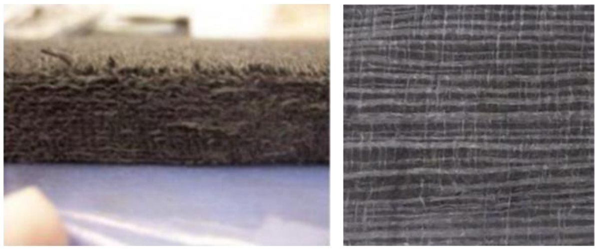

Fig. 7. Left: Example of a needled, carbonized, lyocell plate preform. Right: Through-the-thickness view of a needled lyocell-based composite plate.

2.2 Materials fabrication and characterization methods technologies

Five projects are briefly discussed below:

2.2.1 "Novel Functionally-Graded Coating System for Reusable, Very High Temperature Applications"

Allcomp, Inc. is leading this Phase II SBIR project – NASA MSFC will be conducting CNE testing in the 4th quarter of calendar year 2019 timeframe. This Phase II project started on 31 May 2017 and runs through 30 December 2019. The goal of the project is to develop coated carbon-carbon material systems, for nozzle extension use, that are capable of operating at extremely high temperatures in oxidative environments. Specifically, Allcomp aims to demonstrate systems capable of multi-cycle usage up to at least 4000°F (2204°C) with minimal or no erosion through oxygen/hydrogen engine tests at the Marshall Space Flight Center. Allcomp has conducted multiple rounds of coating development and optimization using their functionally-graded ceramic coating technology, which involves the use of refractory carbide and oxide constituents (see Fig. 8). The use of a functionally-graded coating system enables a significant reduction in the stresses associated with the coefficient of thermal expansion mismatch between the CNE substrate and the coating system constituents. In July 2018, four nozzle extensions were delivered for testing under a MSFC-led liquid oxygen (LOX) / methane (CH_4) lander propulsion project, enabling comparisons with the SBIR's primary LOX / hydrogen (H_2) testing goals. The LOX/methane testing was conducted in the fall of 2018 (see Section 3, below). The targeted applications for this effort are propulsion systems for liquid upper stage and lander descent/ascent engines.

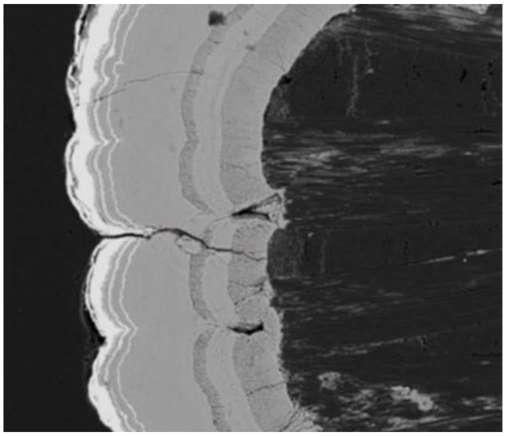

Fig. 8. Early version of Allcomp functionally graded coating on a C-C substrate. The scanning electron microscopy (SEM) photomicrograph illustrates the layer nature of the coating system.

2.2.2 "Nano Enhanced 4000°F CMC for Multiple Use Applications"

Allcomp, Inc. is also leading this recently awarded Phase II SBIR project. This Phase II project started on 14 August 2019 and runs through 13 August 2021. As with the project presented in Sec. 2.2.1, NASA MSFC will be conducting CNE testing to evaluate the effectiveness of the composite material protection scheme presently being developed – testing is to occur in the 2020-2021 timeframe. Typical oxidation protection of C-C make use of internal inhibitors, external coatings, and glass sealants. The goal of this project, which offers the potential for a total paradigm shift in the oxidation protection of C-C composites, is to eliminate the need for an external protection system for CNE's. The aim is to provide molecular level inhibition of both the intra-tow and inter-tow matrix material, thus protecting the carbon fibers by enabling oxidation protection at both the micro- and nano-scale levels.

2.2.3 "Rapid Fabrication of CMCs (Ceramic Matrix Composites) with Aligned Discontinuous Reinforcement via Reactive Melt Infiltration (RMI)"

Materials Research and Design, Inc. is leading this new Phase I SBIR project, which runs from 19 August 2019 through 18 February 2020. The project is being funded under the SBIR Z9.01 "Small Launch Vehicle Technologies and Demonstrations" Subtopic, which addresses the need for more effective vehicle structures and components to enable significant improvements in small launch vehicle affordability. The MR&D project will focus on carbon-carbon materials with oxidation resistance provided via reactive melt infiltration with Group IV A/B metals, such as silicon, zirconium, and hafnium. Material systems such as this have been successfully demonstrated over extended durations in high-temperature oxidizing environments, but currently, there is a lack of understanding regarding how such materials can be optimally processed. To date, manufacturers have implemented a prototyping approach that has been sufficient for the manufacture of demonstration hardware. However, there exists a need for a better understanding of how such materials behave during processing. A program emphasizing multi-scale modeling and simulation of these materials through manufacturing and operational environments will seek to establish a relationship between a variety of process parameters and the quality of the resulting composite material.

2.2.4 "Composite Nozzle Extension Tests Supporting Upper Stage, In-Space, and Lander Propulsion System Development"

As part of the overall effort to develop viable domestic sources for CNE's, MSFC is investigating the following: (a) material property variability within individual nozzle extensions; (b) differences in material property test results obtained from flat panels and from complex shapes; and (c) general material property test data scatter. Southern Research (Birmingham, Alabama) has conducted hoop tension, axial compression, interlaminar tension (ILT), and hoop thermal expansion tests using two tag-end rings excised from the two large Carbon-Carbon Advanced Technologies (C-CAT) nozzle extensions (see Fig. 9), which were subsequently hot-fire tested at MSFC with an oxygen/hydrogen engine (see Sec. 3, below). The tag-end rings were removed from the aft ends of the CNE's such that the forward ends of the tag-end rings were approximately 25 in. in diameter. Both tag-end rings were C-C materials, one lyocell-based and the other polyacrylonitrile- (PAN-) based. The lyocell-based C-C yielded results very similar to those of a rayon-like C-C and demonstrated very low scatter for each type of test conducted [11,12]. The PAN-based C-C test results were also as expected for a PAN-based material, except that there was more data scatter and lower ILT values than expected. As anticipated, the lyocell C-C has a significantly lower axial compression modulus than the PAN C-C, by almost a factor of three. The axial compression strengths for the two materials were fairly similar, with the PAN material being slightly stronger. Overall, the lyocell C-C exhibited less scatter/variability than the PAN C-C. However, it should be noted that all of these test results were obtained from one test article for each material, thus the variability from part to part for each material is unknown and needs to be further assessed if either of these C-C materials are to be used for liquid rocket engines. These results highlight the need for developing databases based upon component hardware and not just flat panels, which often tend to yield overly optimistic results/predictions.

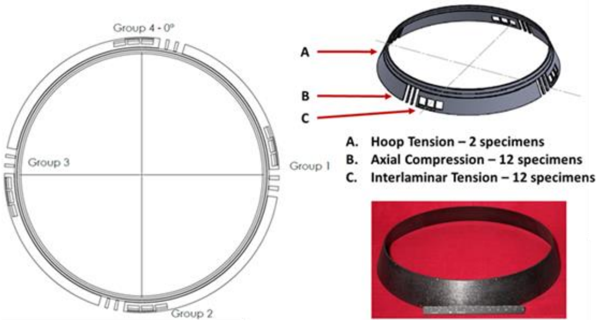

Fig. 9. Cutting plans and photograph of the PAN-based C-C tag-end ring. In order to assess data scatter within the part, groups of three specimens each were excised at four different locations 90° apart around the ring's circumference.

2.2.5 "Additively Manufactured Ceramic Rocket Engine Components"

HRL Laboratories, LLC is leading this public-private partnership project with their subcontractor Vector Launch, Inc. HRL was funded in part through Topic 1 of the NASA Space Technology Mission Directorate 2016 "Small Launch Vehicle Technology Development" Tipping Point solicitation. This Tipping

Point project started on 1 July 2017 and runs through 30 December 2019. The goals of the project are to develop additive manufacturing (AM) technology for reinforced ceramic rocket engine components to enable a factor of 10 reduction in cost, a factor of 10 decrease in fabrication time, and a 10% performance increase versus the state-of-the-art. Using pre-ceramic resins, the HRL technique combines the ease, flexibility, and low cost of polymer additive manufacturing with the high-temperature capabilities of ceramics, thus enabling new designs at lower costs and reduced lead times [13]. Oxygen-methane engine tests have been conducted at Vector Launch facilities (see Fig. 10).

Fig. 11. Left: Subscale 3D printed reinforced pre-ceramic polymer combustion chamber / nozzle component. Center: Same component after matrix pyrolysis. Right: Hot-fire test at Vector Launch.

3. Liquid Engine Test Activities Facilitating CNE Development at the NASA MSFC

Liquid rocket engine technology development projects and on-site test activities at the NASA Marshall Space Flight Center over the past five years have greatly facilitated the development of uncooled composite nozzle extensions. Working with the composite materials and liquid propulsion industries, significant progress has been made (a) in maturing existing materials, and (b) in investigating higher performance materials for use at temperatures up to 4250°F (2343°C). While most of the work to date has made use of a small 1.2K-lb_f thrust engine, two larger engines are also being employed to evaluate larger nozzle extensions. Propellants utilized have included liquid oxygen / gaseous hydrogen (LOX/GH_2), LOX / liquid hydrogen (LOX/LH_2), LOX / liquid methane (LOX/LCH_4), and LOX/kerosene (LOX/RP-1). Engine test activities at MSFC have included the following, each of which is described below:

- Materials screening via 1.2K-lb_f LOX/GH_2, LOX/LCH_4, LOX/RP-1 engine testing,
- Mid-scale demonstration via META4X4 LOX/LCH_4 engine testing, and
- Large-scale demonstration via 35K-lb_f LOX/LH_2 engine testing.

3.1 Small-scale CNE materials evaluation and screening via 1.2K-lb_f thrust engine

Testing with this 1.2K-lb_f thrust engine, which has made use of LOX/GH_2, LOX/LCH_4, LOX/RP-1 propellants, is aimed at assessing various CNE concepts from multiple suppliers. The screening effort is assessing fabrication methods and material systems through a variety of nondestructive evaluation (NDE) techniques, test environments, and pre- and post-test characterization work. The materials evaluated to date have included a range of material systems that have made use of a variety of coatings, matrix chemistries, and attachment concepts. To date, CNE's from the following suppliers have been tested with this 1.2K engine using MSFC's Test Stand 115 (TS-115) facility:

- Allcomp, Inc. – City of Industry, California
- Carbon-Carbon Advanced Technologies, Inc. (C-CAT) – Kennedale, Texas
- Northrop Grumman Innovation Systems (NGIS) – Magna and Promontory, Utah
- Physical Sciences Incorporated (PSI) – Andover, Massachusetts

3.1.1 Establishment of 1.2K-lb_f thrust engine test facility at MSFC TS-115 and LOX/GH_2 testing

The Marshall Space Flight Center created a subscale nozzle test rig in 2014 in order to offer affordable, long duration, hot-fire testing for the screening and evaluation of CNE material systems. Gradl and Valentine have described this test facility and the initial test campaigns using LOX/GH_2 propellants in detail, previously [10]. The facility and testing of NGIS and C-CAT nozzle extensions are briefly summarized, below.

As noted in the referenced American Institute of Aeronautics and Astronautics (AIAA) paper [10], the thruster used on this test rig uses a simple, coaxial injector supplied with LOX/GH_2 to create high temperature environments. The combustion chamber is designed to enable testing of both regeneratively-cooled nozzles and radiatively-cooled nozzle extensions. The target CNE size is approximately 6 in. for length and 5.5 in. for aft-end diameter. The full length contour of the nozzle extension is optimized to an area ratio of 27:1. An attachment area ratio (AR) of approximately 4.4:1 is used, which balances the heat flux on the nozzle and the extension and also maximizes the length of the nozzle extension. The contour is designed to test at sea level conditions. The full AR at the aft end of the nozzle extension is expanded to 27:1 with a wall pressure of 5.5 psia, assuming a chamber pressure, Pc, of 750 psia.

However, the combustion chambers were designed for a maximum Pc of 1350 psia, which allows for future testing of longer CNE's, if desired. A graphic of the thruster with the nozzle extension attached is shown in Fig. 11. Flange attachment configurations have varied depending on the specific CNE material being tested. The test rig enables hot-fire test durations of up to 210 sec.

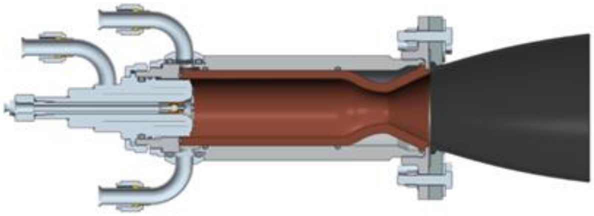

Fig. 11. 1.2K-lb$_f$ thruster with additively-manufactured liner and composite nozzle extension

Composite nozzle extensions provided by NGIS and C-CAT were successfully tested in LOX/GH$_2$ environments using this 1.2K-lb$_f$ engine at TS-115 during the 2014-2016 timeframe. A total of 43 tests were completed in order to characterize the composite substrate materials and coating systems. The primary goal of testing was to enable hot gas exposure of the composite nozzle extensions at temperatures in the 2400-3500+°F (1316-1927°C) range for extended durations. Chamber pressures of 550-750 psig and mixture ratios of 3.5 to 5.9 were utilized. All of the NGIS materials were carbon-fiber tape-wrapped composites that included various combinations of fillers, matrices, and coatings [10]. The C-CAT materials were all based upon their advanced carbon-carbon (ACC) fabrication approach, and included uncoated C-C, SiC pack-cementation coated C-C, and enhanced-matrix versions of their ACC-6 (advanced carbon-carbon densified six times) material [10]. Two photos of representative CNE's are shown in Fig. 12. Additional CNE testing in LOX/GH$_2$ environments is scheduled to begin in the latter part of 2019, primarily in support of the development of lunar lander propulsion systems.

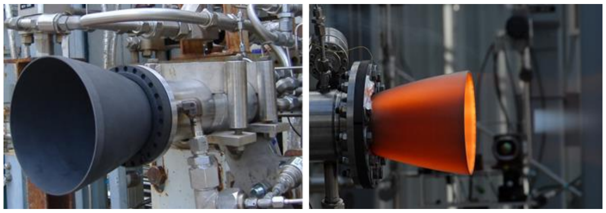

Fig. 12. Left: NGIS nozzle extension installed in Test Stand 115 prior to testing. Right: C-CAT nozzle extension during testing. The C-CAT extension shown has been tested for the longest time to date, a total of 2050 sec. over multiple engine firings.

3.1.2 Use of 1.2K-lb$_f$ thrust engine test facility at MSFC TS-115 for LOX/LCH$_4$ testing

A series of oxygen-methane tests were conducted with high temperature composite nozzle extensions which were manufactured using carbon-carbon (C-C) and carbon/silicon-carbide (C-SiC) fabrication techniques. Testing was performed in an oxygen-methane environment in the September-November 2018 timeframe using much of the same engine test facility hardware employed for the previous LOX/GH$_2$ tests campaigns, described above (Sec. 3.1.1 above, Fig. 13, below) [14]. Hot-fire testing was aimed at identifying materials that could be considered as candidates for lunar lander cryogenic engine configurations – further evaluation and testing is still needed. The thrust chamber assemblies used for these tests included two different chamber configurations with three different injectors.

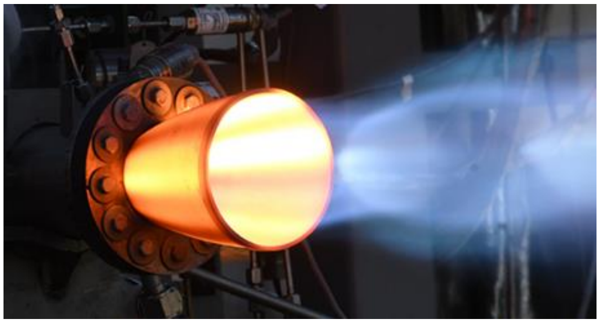

Fig. 13. NGIS nozzle extension with aft-end stiffener during LOX/LCH$_4$ testing.

A total of seven high temperature CNE's were tested from three different composite fabricators, using chamber pressures of 508–910 psig and mixture ratios of 2.43–3.69. A total test time of 2,210 seconds and 20 starts were accumulated collectively with these various nozzle extensions. Individual engine firing durations ranged from 5 to 240 sec. Varying degrees of success were achieved. Some of the CNE's exhibited significant erosion, potentially caused by streaking induced by the injector (see Fig. 14). Varying levels of oxidation were observed on most of the nozzle extensions, although it did not appear any more severe than prior testing in LOX/GH$_2$ environments. The oxidation was observed on both the internal and external surfaces. A few nozzles survived multiple tests and significant time at temperature was accumulated on these units.

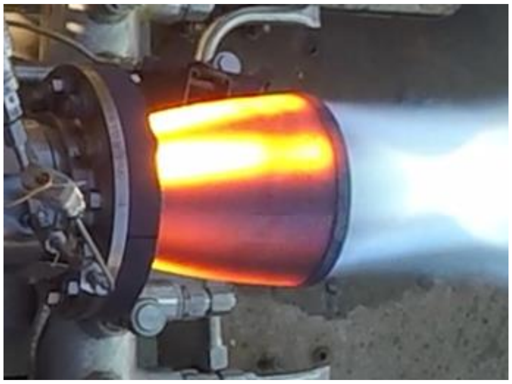

Fig. 14. Video image of hot spots and streaking resulting from shear coaxial injector. This example of a run with an NGIS extension shows much more pronounced streaking than that shown in Fig. 14.

It should be noted that oxidation of the external surfaces was severe on some of the nozzle extensions due to entrainment flow from cryogenic vapors from the facility lines. Although most composite nozzle extensions are designed for altitude operation, ground testing conditions with full length or truncated nozzles should still be evaluated. External oxidation appears to be just as severe as internal oxidation of the composite materials based on visual observations from this test series. A directed inert-gas purge could be used on the external surface, but may not be fully representative of altitude configuration if any free oxygen still exists. Ground testing represents the most conservative test method for external surface oxidation.

As mentioned above, the high temperature CNE's were subject to streaking from the injector, resulting in non-uniform heating and significant circumferential thermal gradients across localized regions. These thermal gradients can cause excessive oxidation and ablation, resulting in burn through, spallation, and/or loss of plies (see Fig. 15). This streaking phenomena should be accounted for in the design of composite nozzle extensions as something beyond just nominal conditions. Additional thermal gradients through the thickness (radially) should also be considered as part of the design and can account for similar failure modes.

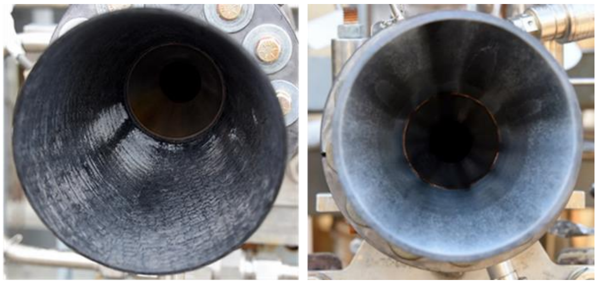

Fig. 15. Two examples of injector-induced streaking effects. The left image shows an Allcomp C-SiC nozzle extension (Part #G12A-034-2), while the right image shows an NGIS C-C nozzle extension (Part #1). Discoloration due to streaking is evident in both images.

Given below is a listing of the seven CNE's tested, as well as an overview of the test conditions and results:

- Allcomp Part #030618-1 – 2D (two directionally-reinforced) C-C, with carbon chemical vapor densification (CVI) and SiC chemical vapor reaction (CVR) matrix, followed by mixed refractory carbide coating.
    - Total hot-fire time = 60 sec.; Number of runs = 1; Maximum temperature = 3386°F (1863°C)
    - Results: Burn through ~ 30 sec. into test at 1-3 o'clock position due to injector streaking.
- Allcomp Part #050918-1 – 2D C-C, refractory metal based matrix inhibition system, with by carbon CVI and SiC CVR matrix, followed by mixed refractory carbide coating.
    - Total hot-fire time = 240 sec.; Number of runs = 1; Maximum temperature = 3369°F (1854°C)
    - Results: Burn through ~ 125 sec. into test at 1-3 o'clock position due to injector streaking.
- Allcomp Part #G12A-034-2 – 2.5D C-SiC, SiC polymer infiltration and pyrolysis (PIP) densification, no external coating
    - Total hot-fire time = 270 sec.; Number of runs = 3; Maximum temperature = 3411°F (1877°C)
    - Results: Minor oxidation during 1st run; burn through during 3rd test at 12-3 o'clock position.
- Allcomp Part #G12A-035-2 – 2.5D C-SiC, SiC polymer infiltration and pyrolysis (PIP) densification, followed by mixed refractory carbide coating.
    - Total hot-fire time = 30 sec.; Number of runs = 1; Maximum temperature = not available
    - Results: Burn through at 7-8 o'clock position at forward end; heavy oxidation on all surfaces.
- C-CAT (no part number, only 1 part) – 2D C-C, with SiC filler in phenolic-based matrix, no external coating.
    - Total hot-fire time = 20 sec.; Number of runs = 2; Maximum temperature = not available
    - Results: Some inner-surface ply delaminations; minor oxidation on all surfaces.

- NGIS Part #1 – 2D C-C, stretch-broken PAN-based prepreg type #1, with mixed carbon phenolic and silicon melt infiltration (MI) based matrix, no external coating. MI performed by Exothermics.
    - Total hot-fire time = 1,180 sec.; Number of runs = 10; Maximum temperature = 3452°F (1900°C)
    - Results: Discoloration on internal and external surfaces; CNE still in excellent condition.
- NGIS Part #2 – 2D C-C, stretch-broken PAN-based prepreg type #2, with mixed carbon phenolic and silicon melt infiltration (MI) based matrix, no external coating. MI performed by Exothermics.
    - Total hot-fire time = 420 sec.; Number of runs = 3; Maximum temperature = 3466°F (1908°C)
    - Results: Discoloration on all surfaces; burn through at forward end due to injector streaking.

3.2 Mid-scale nozzle extension fabrication and up-coming testing via META4X4 7K-lb$_f$ LOX/LCH$_4$ engine

Tentatively planned for late 2019, a mid-size composite nozzle extension fabricated by Carbon-Carbon Advanced Technologies will be tested using an oxygen-methane engine. The CNE was fabricated by C-CAT using their ACC-6 fabrication technology with SiC-filled phenolic resin (i.e., SiC-enhanced C-C). The component has an axial length of 6.45 in. and an aft-end inside diameter of 10.39 in. (see Fig. 16). The technology is of interest for in-space and lander applications.

The MSFC engine being employed for testing is the META4X4, which uses LOX/LCH$_4$ propellants [15]. The engine is based upon MSFC's additively-manufactured META4 engine (Methane Engine Thrust Assembly for 4K-lb$_f$ thrust) that began testing in 2017. The META4X4 engine has a scaled-down version of the META4 combustion chamber (30% smaller diameter), but still offers the same level of thrust.

Fig. 16. Carbon-Carbon Advanced Technologies (C-CAT) SiC-enhanced C-C.

3.3 Large-scale nozzle extension fabrication and test testing via 35K-lb$_f$ LOX/LH$_2$ engine

Carbon-Carbon Advanced Technologies fabricated two upper-stage composite nozzle extensions for testing with the MSFC Low Cost Upper Stage Propulsion (LCUSP) 35K-lb$_f$ thrust oxygen-hydrogen engine [10,15,16]. The CNE's, which both had 25-inch aft-end diameters, were fabricated from two different types of C-C composites: (1) a PAN-based ACC-6 SiC conversion-coated C-C nozzle extension, and (2) a lyocell-based C-C nozzle extension. One made use of polyacrylonitrile-(PAN-) derived carbon fiber and the other made use of cellulosic lyocell-derived carbon fiber. The PAN-based C-C is one of the more widely used types of C-C for current aerospace applications, and is similar to the C-C control surfaces material developed for the MSFC-led X-37 orbital vehicle program and to materials of interest to liquid rocket engine companies for upper stages. The lyocell-based C-C is still very much a research-type material that was first fabricated under a MSFC-led STTR project. Lyocell-based C-C is of interest because it offers the ability to accommodate strains, more readily than PAN-based C-C, when attached to metallic components, as its coefficient of thermal expansion is high for a C-C and its moduli are low. In order to better understand the properties and capabilities of these CNE's, the components were manufactured to axial lengths longer than required for hot-fire testing so that tag-end rings could be removed from the aft ends of the extensions. The two tag-end rings were used for a series of tests performed at Southern Research, which were briefly described above in Sec. 2.2.4.

The engine used for testing the CNE's was the MSFC LCUSP bimetallic additively manufactured thrust chamber. The recently completed LCUSP program was aimed at advancing additive manufacturing of the GRCop-84 copper alloy for liquid engine hardware using selective laser melting and application of a bimetallic deposition jacket. The LCUSP program developed a LOX/LH$_2$ chamber to provide high heat fluxes with a chamber pressure of 1400 psig. In order to allow for a C-C extension to be tested in this environment, it had to replace the regeneratively-cooled nozzle in the thrust chamber assembly (TCA) only test series. This was due to the high area ratio of the regen nozzle, which would result in the composite nozzle extension not flowing full if it were integrated at the aft end of the regen nozzle. The regen nozzle area ratio was maximized to allow for sea-level testing without flow separation. The test setup for these C-C nozzle extensions included the LCUSP chamber and injector, a film coolant ring at the aft end of the main combustion chamber, and the C-C nozzle extension attached to the film coolant ring (see Fig. 17). Film cooling was necessary due to the low area ratio attachment point of the nozzle extensions onto the LCUSP chamber.

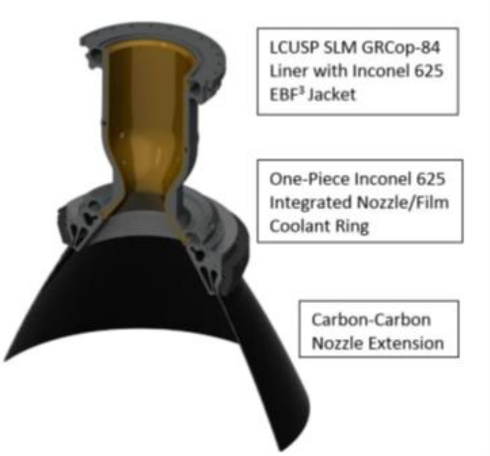

Fig. 17. LCUSP thrust chamber assembly with C-C composite nozzle extension attached.

The two C-C composite nozzle extensions completed testing at MSFC Test Stand 116 (TS-116) in February 2018 (see Fig. 18, Fig. 19). The engine firing (30 sec. at 22K-lb$_f$) with the PAN-based C-C was very successful with no visible damage or degradation and demonstrated an in-process repair of the C-C. The test with the lyocell-based C-C, unfortunately, did not represent a valid test of the material's capability, as it had been damaged either prior to or during installation on the test stand. Both C-C materials are of continuing interest and are being further evaluated under a number of on-going projects.

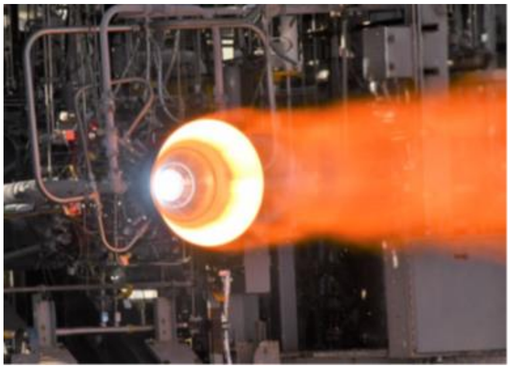

Fig. 18. Test of C-CAT SiC-coated PAN-based C-C with LCUSP engine.

Fig. 19. Two post-firing views of the C-CAT SiC-coated PAN-based C-C.

4. Summary and Conclusions

Extreme-temperature carbon- and ceramic-matrix composite nozzle extensions (CNE's) for liquid rocket engines are currently under development by NASA and its industry partners. A variety of propulsion applications are of interest, including (a) launch vehicle (upper stage) liquid engines, (b) in-space liquid and nuclear thermal propulsion (NTP) systems, and (c) lunar/Mars lander descent/ascent liquid engines. Missions of interest include access to low Earth orbit, the lunar surface, and more distant destinations, such as Mars. More specifically, for NASA, CNE technology development is aimed at supporting the Artemis and Human Landing System Programs, as well as the needs of its Commercial Space partners.

Composite nozzle extensions typically make use of carbon fiber reinforcement architectures in carbon and/or ceramic matrices. Material systems under consideration include carbon-carbon (C-C) and carbon/silicon-carbide (C-SiC) composites, as well as modified versions of such composites that make use of coatings, mixed matrices, and oxidation inhibitors. Development efforts have concentrated primarily on two broad technology areas: (a) materials fabrication and characterization methods, and (b) attachment technology concepts. The expanding interest in using CNE's is due primarily to the following:

- The mass (weight) reductions possible,
- The improved engine performance capabilities that are achievable, especially through the use of higher thermal performance composite materials,
- The potential for significantly reduced costs, as well as
- The rapidly expanding number and variety of potential commercial and exploration missions.

Marshall Space Flight Center efforts have concentrated on (a) further developing the technology and databases needed to enable the use of CNE's on cryogenic liquid rocket engines, (b) developing and demonstrating low-cost capabilities for testing and qualifying such nozzle extensions, and (c) advancing the US domestic supply chain for nozzle extensions. To assist in the advancement of these efforts, hot-fire engine tests of CNE hardware have been conducted with oxygen/hydrogen, oxygen/methane, and oxygen/kerosene propellants using 1.2K-, 7K-, and 35K-lb$_f$ thrust engines at MSFC test stand facilities. These engine tests have enabled the evaluation of both heritage and state-of-the-art material systems, including demonstrating the initial capabilities of new extreme temperature materials.

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